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## ACCELERATED TESTING METHODOLOGY IN CONSTANT STRESS-RATE TESTING FOR ADVANCED STRUCTURAL CERAMICS – A PRELOADING TECHNIQUE

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### ABSTRACT

Preloading technique was used as a means of an accelerated testing methodology in constant stress-rate ("dynamic fatigue") testing for two different brittle materials. The theory developed previously for fatigue strength as a function of preload was further verified through extensive constant stress-rate testing for glass-ceramic and CRT glass in room-temperature distilled water. The preloading technique was also used in this study to identify the prevailing failure mechanisms at elevated temperatures, particularly at lower test rates in which a series of mechanisms would be associated simultaneously with material failure, resulting in significant strength increase or decrease. Two different advanced ceramics including SiC whisker-reinforced composite silicon nitride and 96 wt% alumina were used at elevated temperatures. It was found that the preloading technique can be used as an additional tool to pinpoint the dominant failure mechanism that is associated with such a phenomenon of considerable strength increase or decrease.

### INTRODUCTION

Constant stress-rate (or called "dynamic fatigue") testing has been used to characterize the slow crack growth behavior of advanced ceramics and glass at both ambient and elevated temperatures. The advantage of such testing over other methods lies in its simplicity: Strength is measured in a routine manner at four or more test rates by applying constant displacement rate or constant loading rate. The slow-crack-growth (SCG) or life-prediction parameters required for component design can be estimated from a simple relationship between fatigue strength and applied test rate. These merits have prompted the establishment of ASTM test standards both at ambient (ASTM C1368 [1]) and at elevated (ASTM C1465 [2]) temperatures to determine SCG parameters of advanced ceramics and brittle materials.

A preloading (or accelerated testing) technique has been developed to save test time in constant stress-rate testing at both ambient and elevated temperatures. A certain amount of preload can be applied to the test specimen quickly, see Figure 1, prior to testing as long as the strength with preload does not differ from the strength with zero

preload, resulting in saving of test time. In the previous papers, the authors developed the analytical solution of fatigue strength as a function of preload for natural flaws [3] as well as indentation induced flaws [4]. The solutions have been verified by constant stress-rate testing using different materials such as alumina, glass, silicon nitrides and silicon carbide at ambient and elevated temperatures [3-6]. It was found that a preload corresponding to 80 % of fatigue strength can be applied to most glass and advanced ceramics that typically exhibit the SCG parameter of  $n \geq 20$ , resulting a dramatic saving (80 %) of test time. This accelerated testing technique has been adopted in the ASTM test standards (C 1368 and C 1465) on constant stress-rate testing.

Unlike ambient or low temperature ( $< 900^\circ\text{C}$ ) testing, elevated-temperature testing encounters in some cases several limiting factors such as creep, crack healing/blunting and/or material deterioration, which limit the applicability of constant stress-rate testing. These limitations occur due to a combination of elevated temperature and slow test rate, and result in an inaccurate SCG evaluation if included in the data analysis [7]. Considerable material deterioration takes place at the lower test rates at higher temperatures, see Figure 2. This occurs as a result of enhanced creep deformation often accompanying microcracks and/or macrocracks on the tensile side of flexure test specimens. This data point, corresponding to an appreciable strength drop, should not be used in the analysis to obtain SCG parameters. If included in the data analysis, it causes an underestimation of SCG parameter. On the contrary, a strength increase with decreasing test rate also occurs at lower test rates, due to presumably crack healing or blunting, see Figure 2b. The data point corresponding to this obvious strength increase should be excluded in the analysis to estimate SCG parameters. The application of preload to test specimens prior to testing certainly reduces total test time so that it is expected that the undesirable nonlinear phenomenon - significant strength decrease or increase at lower test rate - would be minimized by a pertinent preload.

The primary purpose of this study was to continue validating the analytical solution of fatigue strength as a function of preload developed, in order to establish a database through extensive testing

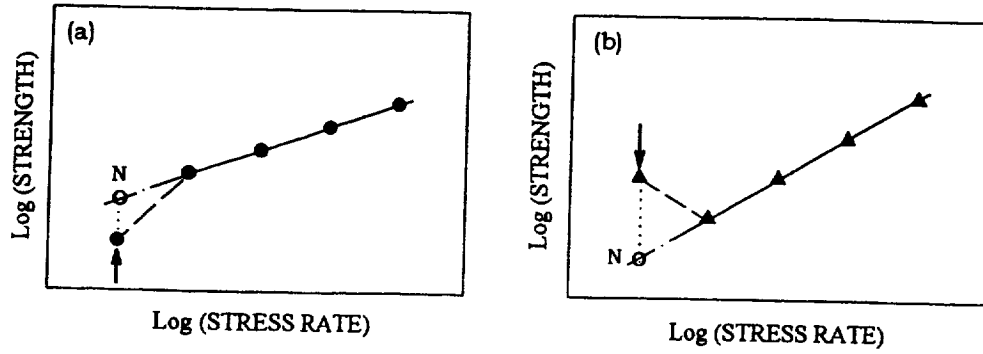


Figure 1. Schematics of significant strength decrease (a) and increase (b) occurring due to different failure mechanisms at lower stress rates in constant stress-rate testing at elevated temperatures. The point "N" represents the 'expected' strength with SCG as a dominant failure mechanism.

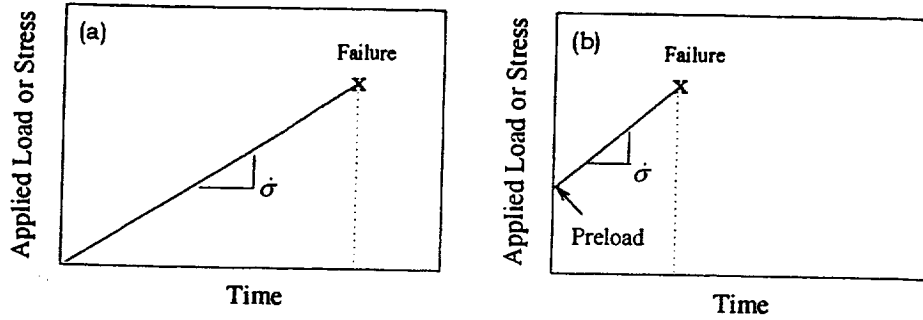


Figure 2. Modes of loading applied in constant stress-rate testing with: (a) no preload (conventional testing), and (b) preload.

with glass-ceramic and CRT (Cathode-Ray Tube) glass at room temperature. The secondary purpose was to better understand how the preloading technique can be used to minimize the phenomenon such as significant strength decrease or increase occurring at lower test rates, identifying governing mechanisms associated with elevated-temperature failure of materials. Two different advanced ceramics were used for this purpose, including 30 vol % SiC whisker-reinforced composite silicon nitride and hot-pressed 96wt % alumina.

## BACKGROUNDS

### Constant Stress-Rate ("Dynamic Fatigue") Testing

Constant stress-rate testing has been used to determine the slow crack growth (or called fatigue, delayed failure or subcritical crack growth) behavior of brittle materials in a wide range of temperatures. Strength is routinely determined as a function of applied test (stress) rate for a given environment. The SCG or life prediction parameters required for component design can be estimated from the following relationship between strength and applied stress rate [1,2]

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \quad (1)$$

where  $\sigma_f$  is the fracture (or fatigue) strength,  $\dot{\sigma}$  is the stress rate applied, and  $n$  and  $D$  are SCG parameters to be determined. The SCG parameter  $n$  and  $D$  can be obtained from the slope and intercept, respectively, of a best-fit linear regression analysis of log (individual

fatigue strength) plotted as a function of log (individual stress rate), based on Eq. (1). Equation (1) is the basis for constant stress-rate testing (ASTM C1368 and C 1465) to determine the SCG parameters of advanced ceramics and brittle materials. The basic SCG law used in Eq. (1) was the following empirical power-law relation:

$$v = \frac{da}{dt} = A \left[ \frac{K_I}{K_{IC}} \right]^n \quad (2)$$

where  $v$ ,  $a$ ,  $t$  and  $A$  are crack velocity, crack size, time, and SCG parameter, respectively.  $K_I$  and  $K_{IC}$  are, respectively, stress intensity factor and critical stress intensity factor (fracture toughness) in mode I loading.

### Preloading Theory

In this section, the previously derived analytical solution of fatigue strength as a function of preload is briefly presented. For the natural flaw system with no localized residual stresses, fatigue strength as a function of preload can be expressed [3]

$$\sigma^* = (1 + \alpha_p^{n+1})^{\frac{1}{n+1}} \quad (3)$$

where  $\sigma^*$  is the normalized fatigue strength, in which the fatigue strength with preload is normalized with respect to the fatigue strength with zero preload,  $\alpha_p$  is preloading factor ( $0 \leq \alpha_p \leq 1$ ), in

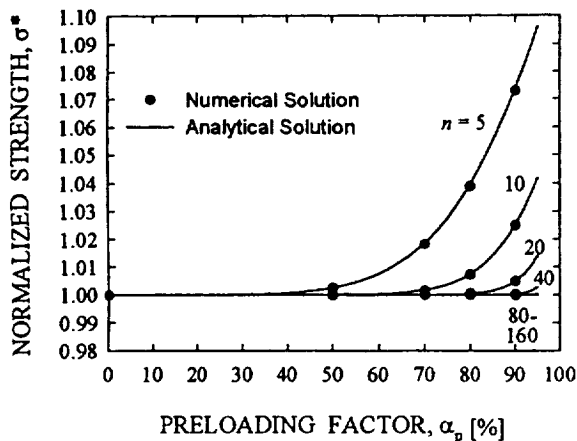


Figure 3. Normalized strength as a function of preloading factor for different SCG parameter  $n$ 's [3].

which the applied preload stress is normalized with respect to the fatigue strength with zero preload. The resulting plot of Eq. (3) for various values of  $n$  is shown in Figure 3. As can be seen in the figure, the normalized fatigue strength increases with increasing preloading factor. The rate of increase in strength is more dominant with decreasing SCG parameter  $n$ , due to enhanced susceptibility to SCG. For most glass and advanced ceramics, the values of  $n$  are typically greater than 20. In this case, a preload corresponding to 90 % of strength ( $\alpha_p = 0.9$ ) results in a strength increase by 0.5 % ( $\sigma^* \leq 1.005$ ), based on Eq. (3). In other words, fatigue strength with a 90 % preload increases by only 0.5 %, compared with the fatigue strength with zero preload. This 90 % preloading gives rise to a 90 % test-time saving, which is the most direct effect of the preloading technique as a means of dramatic saving of test time. An 80 % preloading gives an 80 % test-time saving with a strength difference of only 0.04 % ( $\sigma^* = 1.0004$ ); and so on. A dramatic time saving can be particularly amplified with increasing number of test specimens. Of course, for a material exhibiting a low Weibull modulus ( $m \leq 10$ ), a lower preload should be used to avoid any premature failure, usually with a less-than 80 % preload [6]. Also note that preloading is most effective when applied at the lowest test rate since most of total test time, 80 %, is consumed at the lowest test rate [3].

## EXPERIMENTAL PROCEDURES

### Ambient-Temperature Testing for Glass-Ceramic and CRT Glass

Constant stress-rate testing was conducted for two materials, glass-ceramic and CRT (Cathode-Ray Tube) glass, in ambient-temperature distilled water. Glass-ceramic test specimens were subjected to four-point uniaxial flexure with 20-mm inner and 40-mm outer spans, whereas CRT glass test specimens were subjected to both four-point uniaxial flexure with 40-mm inner and 80-mm outer spans and ring-on-ring biaxial flexure with 22-mm loading ring diameter and 60-mm

support ring diameters. The nominal dimensions of glass-ceramic rectangular beam specimens in uniaxial flexure were 5.0 mm by 2.5 mm by 50 mm, respectively, in width, depth and overall length, while the respective nominal dimensions of CRT glass rectangular beam test specimens were 10 mm by 6 mm by 45 mm. The nominal dimensions of CRT glass disk specimens in ring-on-ring biaxial flexure were 65 mm by 2.5 mm, respectively, in diameter and thickness. The glass-ceramic test specimens were chemically etched to reduce machining damage. The CRT glass specimens were machined, polished and then abraded on the tensile side with sand paper. A total of six different stress rates ranging from 70 to  $7 \times 10^{-4}$  MPa/s were used for glass-ceramic, with a total of 20-30 test specimens at each stress rate. For CRT glass test specimens, a total of five different stress rates ranging from 5.5 to  $5.5 \times 10^{-4}$  MPa/s were utilized in both uniaxial and biaxial flexure, with approximately 23 test specimens at each stress rate. At lower stress rates, some preloads, equivalent to 60 to 95 % of fracture load, depending on material and stress rate, were applied using a quick loading rate (see Figure 1) to the test specimens prior to testing. Preloads were applied for glass-ceramic at two different stress rates of  $7 \times 10^{-3}$  and  $7 \times 10^{-4}$  MPa/s, while for CRT glass they were employed at the lowest stress rate of  $5.5 \times 10^{-4}$  MPa/s. Additional preloading testing was also performed for glass-ceramic at an extremely low stress rate of  $7 \times 10^{-5}$  MPa/s with a 95 % preload, using a total of 14 test specimens. The amount of preload at this lowest test rate was determined based on the 'expected' strength with zero preload, by extrapolating the strength/stress-rate regression data (i.e., Eq. (1)) obtained in a range of 70 to  $7 \times 10^{-4}$  MPa/s. Testing was carried out in accordance with ASTM C 1368 using electromechanical and servohydraulic test frames (Models 8501 and 8562, Instron, Canton, MA) in load control.

### Elevated-Temperature Testing: Identification of Failure Mechanisms

Constant stress-rate testing was previously carried out in flexure in air for 30 vol % SiC whisker-reinforced (GN10) composite silicon nitride at 1300°C [8] and 96 wt % alumina at 1000°C [7]. The detailed experimental procedures, materials and experimental results can be found elsewhere [7,8]. GN10 composite silicon nitride exhibited a decrease in strength at 0.033 MPa/s. Constant stress-rate testing in this work was conducted in flexure for GN10 composite silicon nitride at 0.033 MPa/s where strength decrease occurred. A total of seven specimens were tested at 0.033 MPa/s at 1300°C with an 80 % preload. For alumina, strength with no preload was determined at 0.00033 MPa/s at 1000°C using a total of seven specimens. The strength of alumina with a preload of 90 % was determined at the same stress rate using a total of eight specimens. The amount of preload to be applied was calculated based on the 'expected' strength with zero preload at the stress rate of interest by extrapolating the linear regression line using Eq. (1) together with the SCG parameter ( $n$ ) determined for each material, as follows:

$$P = \alpha_p \sigma_e \quad (4)$$

where  $P$  is the preload to be applied (MPa), and  $\sigma_e$  is the 'expected' strength estimated at the stress rate of interest (e.g., the Point 'N' in Figure 2) by extrapolation of the linear regression line (Eq. (1)) based on the rest of strength data (excluding Point 'N'). All testing was performed using an electromechanical test frame (Model 8562, Instron, Canton, MA) in load-control. Limited fractographic analysis was conducted for selected test specimens to further verify failure mechanisms.

## RESULTS AND DISCUSSION

### Ambient-Temperature Testing

The overall results of constant stress-rate testing for glass-ceramic and CRT glass are shown in Figure 4, where fatigue strength was plotted as a function of applied stress rate according to Eq. (1). The decrease in strength with decreasing stress rate was evident for the two materials, which represents the susceptibility to slow crack growth. The SCG parameter  $n$  was determined as  $n = 21$  and 12, respectively, for glass-ceramic and CRT glass (in both uniaxial and biaxial flexure) by a linear regression analysis based on Eq. (1).

The effect of preload on fatigue strength for the two materials is depicted in Figure 5, in which fatigue strength was plotted as a function of preloading factor ( $\alpha_p$ ). Each horizontal line in the figures represents the fatigue strength with zero preload. No significant variation in fatigue strength as a function of preload was observed from the figures for the two materials.

A comparison of the analytical solution of Eq. (3) with the experimental data can be made by determining the experimental values of  $\sigma^*$  through fatigue strength with preload being normalized with respect to fatigue strength with zero preload and by determining the theoretical values of  $\sigma^*$  (Eq. (3)) with the estimated SCG parameter  $n$  for each material. The resulting plots are shown in Figure 6. For glass-ceramic, the scatter is within approximately 5 %, compared with the theory, resulting in good agreement between the experimental data and the theory. For CRT glass, however, the scatter is increased by about 10 %, due to the increased strength scatter inherent to the test specimens. Despite this inherent scatter, agreement between the experimental data and the theory is reasonable (If standard deviation is used instead of individual data, the agreement would 'look' better). It is particularly noted for the result obtained at the extremely slow test rate of  $7 \times 10^{-5}$  MPa/s for glass-ceramic. The 'expected' strength at this lowest stress rate with no preload can be calculated by extrapolating the data in Figure 4a in conjunction with Eq. (1). This predicted strength was very close within 1 % to the actual strength determined with a 95 % preload using a total of 14 test specimens. Each test specimen at this lowest stress rate would fail in about 480 h (20 days) if no preload is applied. By contrast, with an application of a 95 % preload, time to failure was reduced to about 24 h (one day), resulting in a tremendous saving of test time. Hence, it was feasible to generate in 14 days the strength data at the lowest test rate, compared with 280 days if no preload is used, a great impact on testing economy. To the authors' best knowledge, this would be the first data ever generated at this ultra-low test rate in constant stress rate testing for brittle materials. The results/database of the preloading tests determined for both glass-ceramic and CRT glass in this work indicate again that preloading technique can be used with a reasonable accuracy as an accelerated testing methodology in constant stress-rate testing on the basis of the developed theory, Equation (3).

### Elevated-Temperature Testing: Identification of Failure Mechanisms

The results of elevated-temperature constant stress-rate testing for two materials using the preloading technique at lower stress rates are presented in Figure 7, along with the previous data [7,8]. For the composite silicon nitride, the strength determined at the lowest stress rate of 0.033 MPa/s with an 80 % preload was close to the 'expected' strength that was estimated at the same stress rate by extrapolation of the linear regression line. The strength previously determined with zero preload decreased by about 20 %, compared with the 'expected'

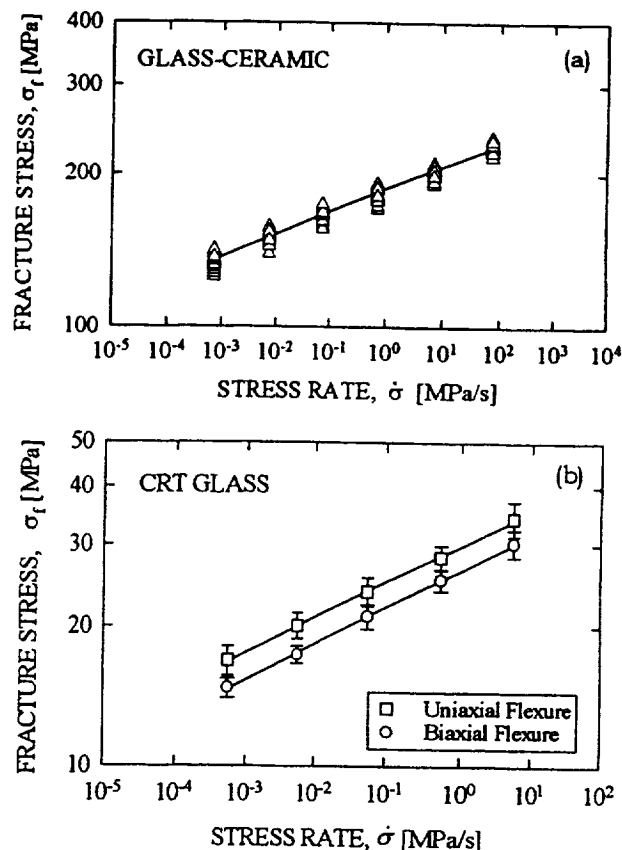


Figure 4. Results of constant stress-rate testing in ambient-temperature distilled water: (a) glass-ceramic in uniaxial flexure; (b) CRT glass in both uniaxial and biaxial (ring-on-ring) flexure. For clarity of presentation, the average strength was plotted for CRT glass (b).

strength [8]. On the contrary, the strength with an 80 % preload decreased by only 7 %. Hence, a significant strength recovery of about 16 % was achieved with the preload up to 80 %. It had been observed that the specimens tested with zero preload exhibited not only an appreciable creep deformation (creep strain  $\approx 0.5$  %) but also the presence of microcracks and macrocracks on the tensile side of the flexure specimens. By contrast, the specimens tested with a preload of 80 % did not exhibit any sign of the presence of micro- or macrocracks on the tensile surface. This indicates that shortened test time - a reduction of 80 % in time - significantly minimized creep deformation, while slow crack growth was operative as a major failure mechanism.

The results for 96wt % alumina both with and without preload are depicted in Figure 7b. The strength with zero preload at the lowest stress rate of 0.00033 MPa/s increased dramatically by about 76 %, compared with the 'expected' strength; whereas the strength with a preload of 90 % was very close to the 'expected' strength with a negligible increase of 3 %. The reduced test time for alumina, due to the preload applied, brought their strength back to the 'expected' strength where slow crack growth would be a governing failure mechanism.

A summary of normalized strength thus obtained with different preloads is presented in Figure 8. Again, the normalized strength was

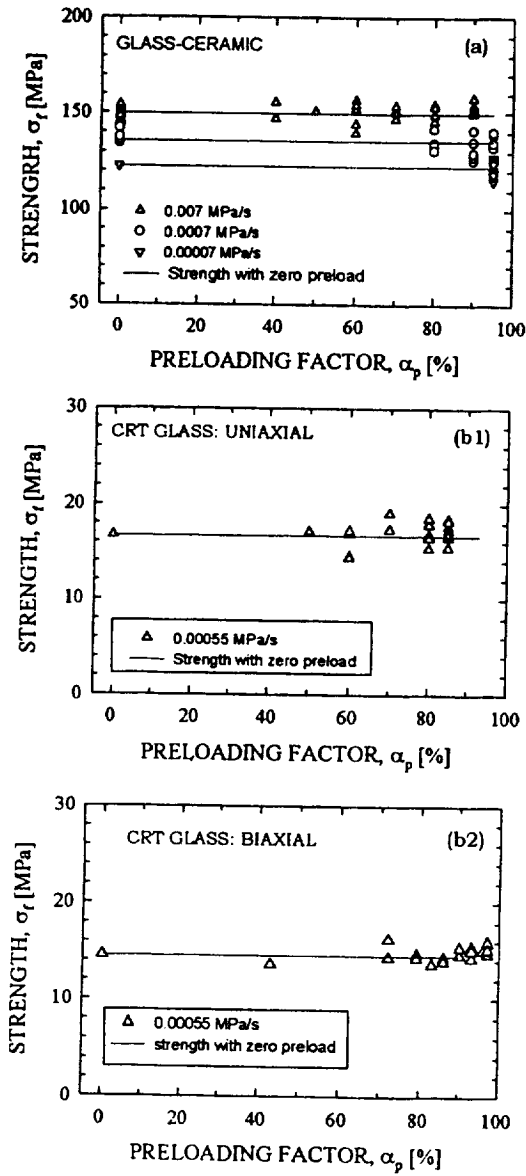


Figure 5. Strength as a function of preload determined in ambient-temperature distilled water: (a) glass-ceramic in uniaxial flexure; (b) CRT glass in both uniaxial and biaxial (ring-on-ring) flexure.

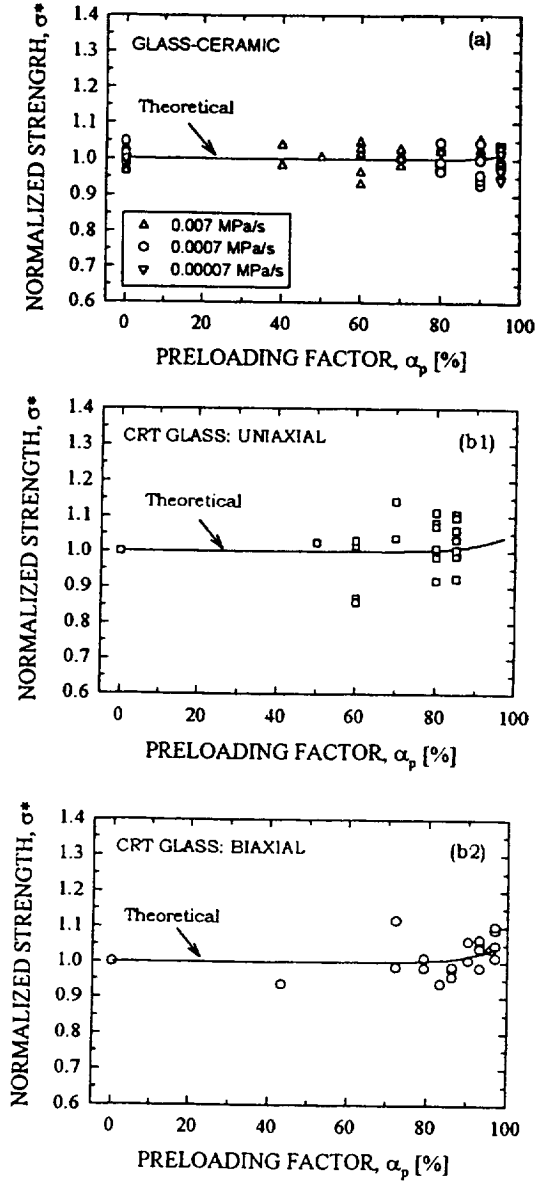


Figure 6. Normalized strength as a function of preload determined in ambient-temperature distilled water: (a) glass-ceramic in uniaxial flexure; (b) CRT glass in both uniaxial and biaxial (ring-on-ring) flexure. The theory based on Eq. (3) is included.

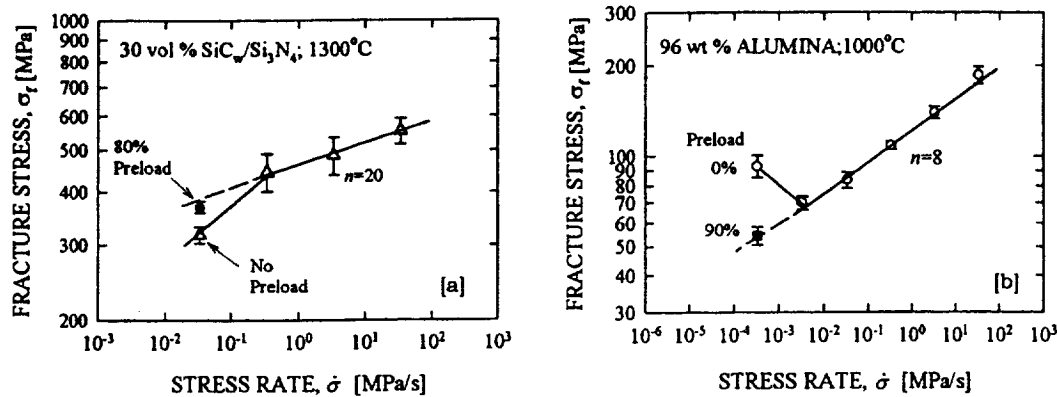


Figure 7. Results of constant stress-rate flexure testing with preloading at lowest test rates for: (a) 30 vol % SiC whisker-reinforced silicon nitride (GN10 composite) with an 80 % preload at 1300°C; (b) 96 wt % alumina with zero and 90 % preloads at 1000°C. The previous results on constant stress-rate testing [7,8] are included for comparison.

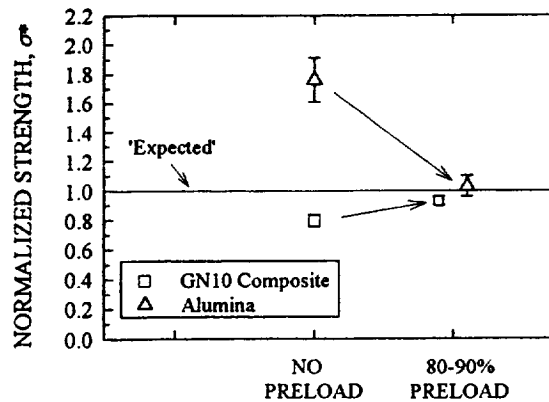


Figure 8. Normalized strength as a function of preload for 30 vol % SiC whisker-reinforced silicon nitride (GN10 composite) at 1300°C and 96 wt % alumina at 1000°C, determined from the strength data at the lowest test rates in Figure 7.

defined as strength with preload, normalized with respect to the 'expected' strength with zero preload in which a governing failure mechanism is slow crack growth. Figure 8 clearly shows that without preloading the strength deviation from the 'expected' strength was obvious, with an increase of 76 % for alumina and with a decrease of 20 % for the composite silicon nitride, respectively. With a preload of 80 or 90 %, the corresponding strength all converged to the 'expected' strength for the two materials. Although not presented here, it was found that alumina exhibited a little strength increase (about 20 %) from the 'expected' strength when a lower preload typically less than 70 % was applied.

All of the elevated-temperature results above indicate that with a preload of 80 or 90 % the resulting strength approached the 'expected' strength with slow crack growth being a dominant mechanism associated with failure. A significant strength decrease [8-10] occurs as a result of enhanced creep deformation typically accompanying

microcracks and/or macrocracks on tensile side. An appreciable strength increase with decreasing stress rates, similar to the phenomenon known as 'strain-rate reverse effect' [11], has been often observed for various silicon nitrides [5,12], silicon carbides [12,13] and alumina [11]. This aspect is understood such that crack healing and/or crack-tip blunting dominates the slow crack growth propagation, resulting in a significant increase in strength at lower stress rates. Preloading to  $\geq 80$  % shifts the failure mechanism out of this crack healing/blunting regime and into the slow crack growth regime, by reducing test time. Therefore, the preloading technique can be used not only to save test time but as an additional tool for pinpointing the dominant failure mechanism(s). In the latter case, one can utilize the preloading technique to maximize the desired SCG mechanism and thus to obtain a true measure of fatigue strength, which is crucially important to determine accurate life prediction parameters in constant stress-rate testing at elevated temperatures.

## CONCLUSIONS

The results of preloading tests determined for both glass-ceramic and CRT glass in room-temperature distilled water indicate again that preloading technique can be used with a reasonable accuracy as an accelerated testing methodology in constant stress-rate testing on the basis of the developed theory. A significant strength increase or decrease, occurring at elevated temperatures at lower stress rates due to crack-healing/crack-tip-blunting or enhanced creep deformation with microcracks or macrocracks, was minimized with an application of preload of 80 to 90 %. The significant strength deviation of 20 to 76 % was reduced to a range of only 3 to 7 %, by maximizing the desirable failure mechanism, slow crack growth, as verified using two different advanced ceramics of 30 vol % SiC whisker-reinforced composite silicon nitride and 96 wt % alumina. Thus, the preloading technique can be used not only as an accelerated testing methodology, but also as an additional tool for pinpointing failure mechanisms of advanced ceramics in constant stress-rate testing at elevated temperatures where a nonlinearity in the log (*fatigue strength*) vs. log (*stress rate*) relation occurs.

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